



Designation: E841 – 04

Standard Test Method for Determination of Copper in Iron Ores and Related Materials by Atomic Absorption Spectrometry¹

This standard is issued under the fixed designation E841; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of copper in iron ores, concentrates, agglomerates, and related materials in the concentration range from 0.003 to 1 %.

1.2 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 *ASTM Standards:*²

[D1193 Specification for Reagent Water](#)

[E50 Practices for Apparatus, Reagents, and Safety Considerations for Chemical Analysis of Metals, Ores, and Related Materials](#)

[E135 Terminology Relating to Analytical Chemistry for Metals, Ores, and Related Materials](#)

[E173 Practice for Conducting Interlaboratory Studies of Methods for Chemical Analysis of Metals](#)³

[E276 Test Method for Particle Size or Screen Analysis at No. 4 \(4.75-mm\) Sieve and Finer for Metal-Bearing Ores and Related Materials](#)

[E663 Practice for Flame Atomic Absorption Analysis](#)³

[E877 Practice for Sampling and Sample Preparation of Iron Ores and Related Materials for Determination of Chemical Composition](#)

[E882 Guide for Accountability and Quality Control in the Chemical Analysis Laboratory](#)

[E1601 Practice for Conducting an Interlaboratory Study to Evaluate the Performance of an Analytical Method](#)

3. Terminology

3.1 *Definitions*—For definitions of terms used in this test method, refer to Terminology [E135](#).

4. Summary of Test Method

4.1 The sample is dissolved in hydrochloric acid with the addition of a small amount of nitric and hydrofluoric acids. After evaporation to fumes with perchloric acid, the solution is diluted with water and filtered. A portion of the solution is examined by atomic absorption spectroscopy using standards containing approximately the same amount of iron as the test sample.

5. Significance and Use

5.1 In the making of iron and steel during the reduction of iron ores, copper forms alloy with iron and steel hence the necessity of determining the copper concentration for metallurgical consideration.

5.2 This test method is intended to be used for compliance with compositional specifications for copper content. It is assumed that all who use these procedures will be trained analysts capable of performing common laboratory procedures skillfully and safely. It is expected that work will be performed in a properly equipped laboratory and that proper waste disposal procedures will be followed. Appropriate quality control practices must be followed, such as those described in Guide [E882](#).

6. Interferences

6.1 None of the elements normally found in iron ores interfere with this test method.

7. Apparatus

7.1 *Atomic Absorption Spectrometer*, meeting the following criteria:

¹ This test method is under the jurisdiction of ASTM Committee [E01](#) on Analytical Chemistry for Metals, Ores, and Related Materials and is the direct responsibility of Subcommittee [E01.02](#) on Ores, Concentrates, and Related Metallurgical Materials.

Current edition approved May 1, 2004. Published June 2004. Originally approved in 1981. Last previous edition approved in 1999 as E841 – 99. DOI: 10.1520/E0841-04.

² For referenced ASTM standards, visit the ASTM website, www.astm.org, or contact ASTM Customer Service at service@astm.org. For *Annual Book of ASTM Standards* volume information, refer to the standard's Document Summary page on the ASTM website.

³ Withdrawn. The last approved version of this historical standard is referenced on www.astm.org.

7.1.1 *Minimum Sensitivity*—The absorbance of the highest calibration solution (see 8.6) must be at least 0.3.

7.1.2 *Curve—Linearity*—The difference between the readings of the two highest calibration solutions must be more than 1.4 times the difference between the readings for the zero solution and the lowest calibration solution (see 8.6).

7.1.3 *Minimum Stability*—The coefficient of variation of a number of measurements of the highest calibration solution and of the zero calibration solution must be less than 1.5 and 0.5 % respectively, relative to the measurement of the highest calibration solution.

NOTE 1—A strip chart recorder or digital readout device, or both is advisable to measure the criteria in 7.1 and for all subsequent measurements.

NOTE 2—A background corrector equipped with a hydrogen or a deuterium hollow cathode lamp is advisable for the concentration range from 0.003 to 0.010 % Cu.

NOTE 3—Instrument parameters will vary with each instrument. The following parameters were successfully used in several laboratories and they can be used as guidelines. Solutions were aspirated into an air-acetylene flame of a premix burner.

Hollow cathode lamp, mA	3
Wavelength, nm	324.7
Air flow-rate, L/min	10
Acetylene flow-rate, L/min	2.5

8. Reagents and Materials

8.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society where such specifications are available⁴. Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

8.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean reagent water as defined by Type I of Specification D1193.

8.3 *Copper Standard Solution A (1 mL = 1.0 mg Cu)*—Dissolve 1.000 g of copper metal (minimum 99.5 %) in 30 mL of nitric acid (1 + 1) and dilute to 1 L.

8.4 *Copper Standard Solution B (1 mL = 0.10 mg Cu)*—Transfer 100 mL of Standard Solution A to a 1-L volumetric flask, dilute to volume, and mix.

8.5 *Copper Standard Solution C (1 mL = 0.01 mg Cu)*—Transfer 100 mL of Standard Solution B to a 1-L volumetric flask, dilute to volume, and mix.

8.6 *Copper Standard Calibration Solution*—Depending on the expected copper concentration in the sample, prepare calibration solution using copper Standard Solution B or C. For the concentration range from 0.01 to 0.2 % Cu, use copper Standard Solution B and for the concentration range from 0.003 to 0.02 %, use copper Standard Solution C.

8.6.1 Transfer 1.0, 3.0, 5.0, 7.0, and 10.0-mL portions of copper Standard Solution B or C to 100-mL volumetric flasks. Add 20.0 mL of background solution, dilute to volume, and mix.

8.7 *Hydrochloric Acid (HCl) (1 + 1)*—Mix 1 volume of hydrochloric acid (HCl) with 1 volume of water.

8.8 *Iron Background Solution*—Dissolve 15 g of high-purity iron metal in 150 mL HCl (1 + 2) and oxidize by the dropwise addition of HNO₃. Add 250 mL of perchloric acid (HClO₄) and evaporate to fumes. Allow to fume for 10 min, cool, and dilute to 1 L.

8.9 *Nitric Acid (HNO₃) (1 + 1)*—Mix 1 volume of nitric acid (HNO₃) with 1 volume of water.

8.10 *Reference Solution*—Transfer 20 mL of the background solution to a 100-mL volumetric flask, dilute to volume, and mix.

9. Hazards

9.1 For precautions to be observed in this method, refer to Practice E50.

10. Sampling and Sample Preparation

10.1 Collect and prepare the test unit in accordance with Practice E877.

10.2 The analytical sample shall be pulverized so that at least 95% passes a No. 100 (150- μ m) sieve in accordance with Test Methods E276.

NOTE 4—To facilitate decomposition, some ores, such as specular hematite, require grinding to pass a No. 200 (75- μ m) sieve.

11. Procedure

11.1 Transfer approximately 0.5 g of the sample to a small weighing bottle previously dried at about 150°C. Dry bottle and contents for 1 h at 105 to 110°C. Cap the bottle and cool to room temperature in a desiccator. Momentarily release the cap to equalize the pressure and weigh the capped bottle and sample to the nearest 0.1 mg. Repeat drying and weighing until there is no further weight loss. Transfer the sample to a 250-mL beaker and reweigh the capped bottle to the nearest 0.1 mg. The difference between the two weights is the weight of the sample taken for analysis.

11.2 Carry a reagent blank through all steps of the procedure starting with 11.3.

11.3 *Decomposition of Sample*—Moisten the sample with a few millilitres of water, add 25 mL of HCl, cover, and digest below the boiling point until no further attack is apparent. Add 5 mL of HNO₃ and heat for 10 min. Remove cover, add 3 mL of hydrofluoric acid, and heat for 10 min. Rinse wall of the beaker with water, add 5 mL of HClO₄, and evaporate slowly to dense white fumes of HClO₄. Fume for 2 to 3 min and allow the solution to cool. Add 50 mL of water and warm until the soluble salts are in solution. Filter the solution through a fine texture paper, wash the residue with warm water, receiving the filtrate and washings in a 100-mL volumetric flask. Ignite the filter paper containing the insoluble residue in a platinum crucible, moisten the residue with a few drops of water, add about 3 mL of hydrofluoric acid and 2 drops of sulfuric acid. Add a few drops of nitric acid. Mix and evaporate to fumes of sulfuric acid. Cool, cautiously add 2 to 3 mL of water to

⁴ *Reagent Chemicals, American Chemical Society Specifications*, American Chemical Society, Washington, DC, www.chemistry.org. For suggestions on the testing of reagents not listed by the American Chemical Society, see the *United States Pharmacopeia and National Formulary*, U.S. Pharmacopeial Convention, Inc. (USPC), Rockville, MD, <http://www.usp.org>.